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TECHNICAL REPORT FRL-TR-38

EPR OBSERVATION OF NH_3^+ FORMED BY X-RAY IRRADIATION OF AMMONIUM PERCHLORATE CRYSTALS

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AUGUST 1961

FELTMAN RESEARCH LABORATORIES
PICATINNY ARSENAL
DOVER, N. J.

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DEPT. OF THE ARMY PROJECT 5504-01-027

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by

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ABSTRACT

An investigation of irradiated ammonium perchlorate was conducted by electron spin resonance. A new specie, the NH_3^+ radical was discovered. This ion radical appears to be stable up to at least 125°C.

INTRODUCTION

Profound changes in the decomposition characteristics or chemical reactivity of ammonium perchlorate crystals following X-ray and gamma ray irradiation have been reported (Refs 1 and 2). In an effort to understand the nature of the radiation damage in these crystals, we have undertaken an investigation using electron paramagnetic resonance (EPR) techniques. One would expect, with these techniques, to observe those damage sites in the crystal which happen to contain unpaired electron spins.

SAMPLE PREPARATION

The ammonium perchlorate crystals used in this work were doubly recrystallized from triply distilled water. The original material was certified reagent grade salt obtained from the Fisher Scientific Company. Both crystalline powders and larger crystals of several cubic millimeters were prepared. The samples were irradiated with an exposure dose of 10^6 roentgens at a rate of 2×10^5 roentgens per hour by means of an OEG-50 X-ray tube with a molybdenum target.

ELECTRON PARAMAGNETIC RESONANCE APPARATUS

The electron paramagnetic resonance experiments were performed using the Varian V4501 EPR spectrometer. This instrument employs 100 kc field modulation and a microwave frequency of 9.5 kmc. A Varian V4547 variable temperature accessory permitted studies over a continuous range of temperature.

RESULTS AND DISCUSSION

Figure 1 shows the EPR spectrum obtained at room temperature from a single crystal of irradiated ammonium perchlorate. The spectrum is what might be expected from hyperfine interaction with one nucleus of spin 1 and three equivalent nuclei of spin $^1/_2$, and almost certainly arises from an unpaired electron spin strongly localized on an NH_3 molecule. Contact with the nitrogen nucleus splits the spectrum into three lines of equal spacing and intensity, and contact with the three equivalent

protons splits each of these three lines into a quartet of equally spaced lines with intensity ratios of 1:3:3:1. This analysis is illustrated in Figure 1. Presumably the radical is a positive ion, although the EPR technique would not distinguish it from a negative ion. No resonances other than that associated with the NH_3^+ radical were found in these crystals.

Several interesting observations can be made concerning the anisotropy and temperature dependence. Figure 2 shows spectra obtained with a powder sample at +100°C, +25°C, -100°C, and -180°C. At +25°C, the spectra from the powder and the single crystal differ markedly, but at +100°C the spectra are nearly the same. We conclude that, at the high temperature, the NH_3^+ molecules are rotating rapidly and the anisotropy which causes the difference in spectra found at lower temperatures is being averaged out. The critical rotation frequency is $\gamma \overline{\Delta H}$, where γ is the gyromagnetic ratio of the electron dipole and $\overline{\Delta H}$ is an average shift in the position of the hyperfine lines because of the anisotropy. The separation of the lines at +100°C is therefore a measure of the isotropic hyperfine splitting. Contact with the nitrogen nucleus splits the lines by 18.1 oersteds and contact with the hydrogen nuclei splits the lines by 25.0 oersteds. The splittings were measured to an accuracy of one per cent by comparing them with the resonance of nitrosodisulphonate (peroxylamine disulphonate) ion $(ON(SO_3)_2)^=$, which is split into three lines 13.0 oersteds apart.

At -100°C (Fig 2), the central quartet corresponding to $M = 0$ for the nitrogen nucleus is well defined and of much greater intensity than the quartets corresponding to $M = \pm 1$. It is quite possible that the observed anisotropy arises from interaction with the quadrupole moment of the nitrogen nucleus, since the nitrogen splittings are so much more drastically affected than are the hydrogen splittings. Hydrogen, of course, has no quadrupole moment. It is also possible that the electron dipole has stronger anisotropic interaction with the dipole moment of the nitrogen nucleus than with the proton dipoles. In any event, the molecule is rotating slowly compared with $(\gamma \overline{\Delta H_N})^{-1}$, where $\overline{\Delta H_N}$ refers to the average anisotropic shift arising from interaction with the nitrogen nucleus. At still lower temperatures (-180°C), the spectrum from the

powder becomes asymmetric with respect to the center of the spectrum (Fig 2), although the single crystal spectrum remains symmetric. Anisotropy arising from interaction between the electron dipole and the proton dipoles enters the picture, and the molecule rotates slowly compared with $(\gamma \overline{\Delta H_p})^{-1}$ where the subscript "P" refers to the protons. Rogers and Pake[3] have reported similar anisotropic effects when the vanadyl ion, VO^{++}, is in aqueous solution. The behavior of the anisotropy of the NH_3^+ resonance as a function of temperature suggests that freedom of the ion to rotate is frozen out in several fairly well defined steps.

The ammonia ion is quite stable with respect to time and temperature. No decay of the resonance intensity was noticed upon heating to +125°C or upon storing for several months at room temperature.

The resonance cannot be saturated at room temperature with the available microwave power (200 mw at the sample cavity); at lower temperatures, however, it is necessary to reduce the power level to avoid saturation. The g value of the center of gravity of the spectrum was measured by comparison with crystalline dpph (g = 2.0036) and found to be 2.0034 ± 0.0001.

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3. R. N. Rogers and G. E. Pake, J. Chem. Phys. 33, 1107 (1960)

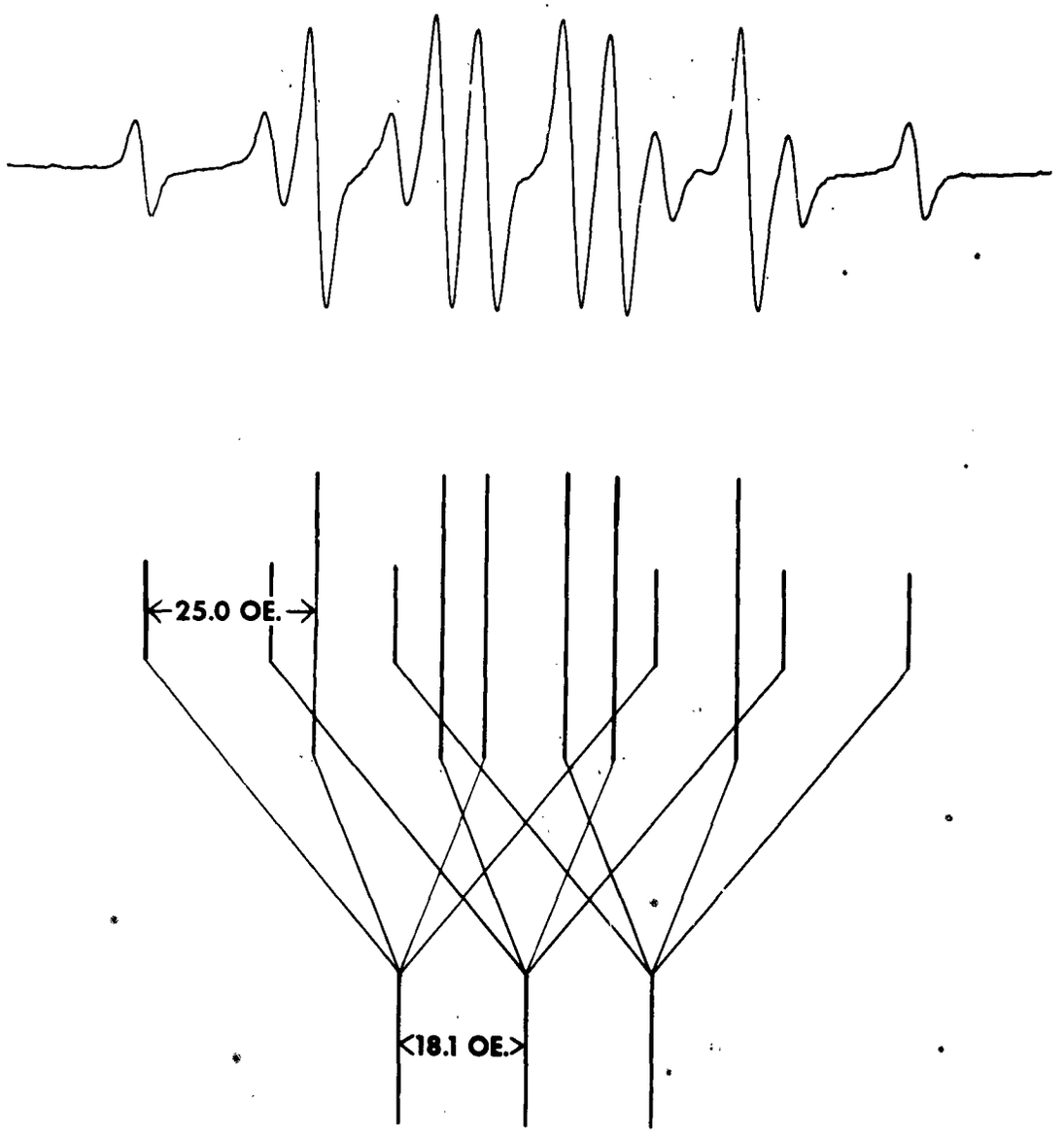

Fig. 1 Room temperature EPR spectrum from a single crystal of irradiated ammonium perchlorate, and the reconstructed spectrum from NH_3^+ assuming isotropic hyperfine interaction

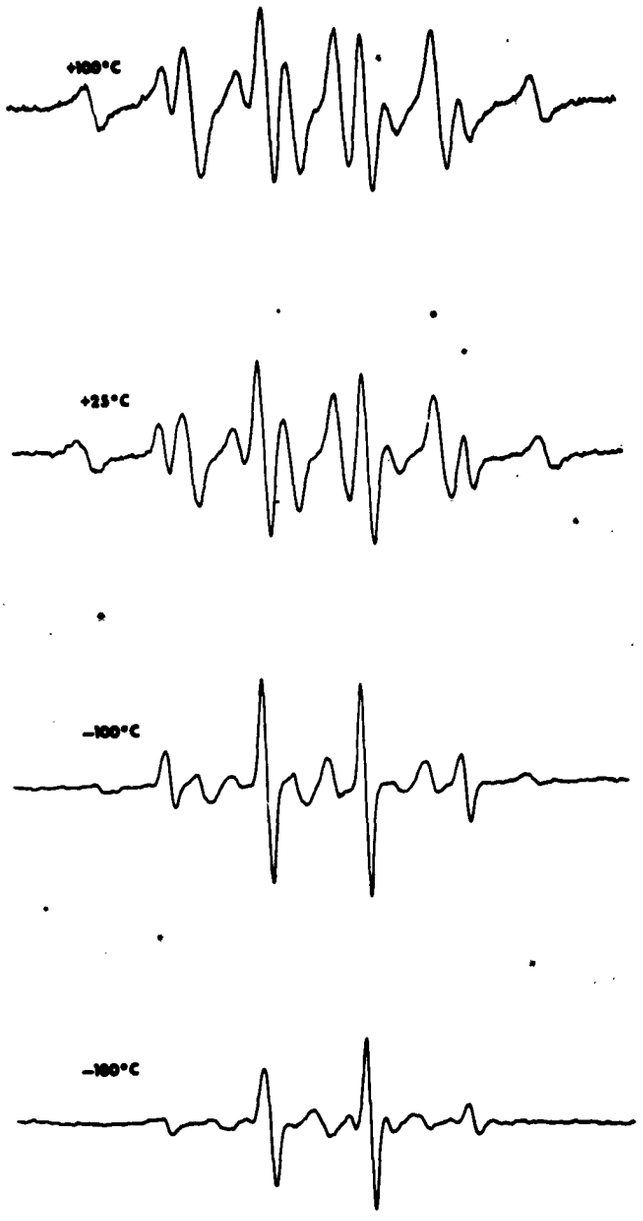

Fig 2 EPR spectra at +100°C, +25°C, -100°C, and -180°C from a crystalline powder of irradiated ammonium perchlorate

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